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National Lubricating Grease Institute Appoints

CARL E. BOLTE, *Executive Secretary*

Announcement has just been made of the appointment of Mr. Carl E. Bolte as Executive Secretary of the National Lubricating Grease Institute, to begin his new duties July 1st.

Mr. Bolte recently returned from a month's stay in the British Isles where he was sent on an Anglo-American mission by the President of Rotary International, of which he is a director. In this capacity he spoke to Rotary, civic, and other groups in England, Ireland, Scotland and Wales. He also conferred with government officials, and business and church leaders concerning postwar problems. A written report of his observations and experiences is being carried serially by several newspapers and magazines.

Mr. Bolte comes to the National Lubricating Grease Institute with a distinguished record of service in business, church and civic enterprises. Most recently he has been the associate director of the American War Dads. Before assuming these duties he had been president and general manager of the Slater Mill & Elevator Co., Slater, Missouri, during which period he also served as president of the Missouri Millers' Association and director of the Millers' National Federation. Mr. Bolte is at present chairman of



the board of directors of the Missouri State Chamber of Commerce, having previously served two years as president of that body. He has been a member of the Missouri State Planning Board. For two years during the war he served as director of the Industrial Service Division, Smaller War Plants Corporation, in Washington, D. C. William Jewell College and the University of Missouri are Mr. Bolte's alma maters. He is active in the alumni association

of the University of Missouri, and also a trustee and member of the executive committee of Missouri Valley College at Marshall, Missouri. For many years he has been an active member of the First Baptist Church at Slater. While a student at the University of Missouri, he was president of the famous Burrall Bible Class which had an average attendance of more than one thousand during his tenure of office. For many years, too, Mr. Bolte was an officer in the Land of the Ozarks Council, Boy Scouts of America.

Appointment of Mr. Bolte as full-time Executive Secretary marks another forward step in the thirteen year history of the National Lubricating Grease Institute. Founded in 1933 for the purpose of developing and disseminating information pertinent to the industry, the N.L.G.I. today includes among its members most of the leading grease manufacturers of the country. Its Technical Committee, in addition to developing the widely-used N.L.G.I. grease classification, has done an immense amount of research and development work, operating independently and also in cooperation with committees from other technical societies, and with governmental and military agencies.



The Effect of Fatty Acid Molecular Weight on Lithium Greases

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The inception of the Earle⁹ patents created a widespread interest in industrial applications for Lithium greases and since these versatile lubricants possessed unique properties, especially adaptable to exacting lubrication problems, their initial field of application was for military aircraft. Such applications demand maintenance of the highest order of quality and uniformity, so a thorough review of the factors affecting the critical properties of a grease was indicated. It soon became evident that a great volume of work had been done to provide the ultimate in lubricating oils, but all too little information was available on grease makers soaps and on lithium soaps in particular. Consequently, the authors set out to investigate the properties of lithium soaps as they affect the greases.

In approaching this investigation, the wide variation in acid contents of "commercial fatty acids" appeared to be a major factor influencing the properties of lithium grease and so this was singled out for investigation. The extent of the variation in "commercial fatty acids" is more readily apparent when Table I is consulted, for even the sharpest cut fractions are composed of little more than half true stearic acid, while the fish oil derivatives contain acids ranging from 14 to 22 carbon atoms in chain lengths. It is therefore obvious that a wide variation in lithium grease is to be expected dependent on the source of the fatty acid used in the production of lithium soap.

GREASE EVALUATION TESTS

To evaluate the effect of these fatty acid variations on the properties of lithium greases, physical tests had to be selected which were both reproducible and closely related to the actual functions of the grease

in industrial equipment. This presented a problem, for most of the physical evaluation tests on greases are in a state of flux; their actual relation to service behavior is obscure and reproducibility is questionable. A survey of the many grease evaluation tests in use led to the final selection of bleeding, penetration and dropping point as reproducible tests, generally acceptable to the industry and closely associated with commercial functions of the lubricant.

The bleeding test is a measure of stability of the grease to separation of oil or syneresis at elevated temperatures. It is evident that a gel which loses too much of its liquid phase, is unstable and not suitable for commercial lubrication.

Penetration¹ is a measure of consistency and is therefore important to the grease manufacturer who uses it as a method for calculating yield of grease from a given soap content. It is also of value in indicating the most suitable application for a given grease, since, as in oils the viscosity determines the use, so in greases the penetration directs the specific application. Delta penetration is a term derived by the authors to define the change in penetration or consistency of grease after application of shearing stresses. Thus a gel which undergoes a great decrease in penetration or has a high Delta value, is not desirable for industrial lubrication. Obviously, a lubricant of sufficient initial consistency designed to function in a bearing of standard design, loses its value if after operation, this consistency is so altered that the grease will either no longer remain in the bearing or becomes so heavy that it impedes proper function of the unit.

Finally, dropping point² was chosen as a control test because it defines the upper

TABLE II

Name of Fatty Acid	Carbon Chain Length
Caprylic	C ₈
Capric	C ₁₀
Lauric	C ₁₂
Myristic	C ₁₄
Palmitic	C ₁₆
Stearic	C ₁₈
Arachidic	C ₂₀
Behenic	C ₂₂
Ricinoleic	C ₁₈ One double bond One hydroxyl
Linoleic	C ₁₈ Two double bonds
Oleic	C ₁₈ One double bond

temperature limit at which a grease may be used as such, since above this point it acts as a fluid rather than a gel.

RAW MATERIALS USED

To adequately cover the wide molecular weight range of the constituent acids comprising commercial fatty acids, it was necessary to prepare lithium salts of pure acids ranging from 8 to 22 carbon atoms in length. These acids, (a detailed list is given in Table II) were purified by the authors so that lithium salts averaging 90% purity were obtained, the remaining 10% consisted of other kindred acids. The preparation of the lithium salt was accomplished by reacting the fatty acid with 99.5% Li OH in an aqueous medium at 95° C. The slurry was then filtered, washed, dried and the water insoluble lithium salts finished to a pH of about 8. These pure lithium soaps were then used to compound the lithium greases were then used to compound the lithium grease.

It is well known that the type and viscosity of the oil used has a marked effect on the characteristics of the grease produced from it. Inasmuch as the variety of available oils suitable for lithium grease making is extensive, the authors chose to use typical medium viscosity paraffinic and naphthenic oils in these experiments. To eliminate the effect of oil additives, such as oxidation inhibitors, viscosity index improvers, pour point depressors, etc., only oils of the type specified in Table III were used.

TABLE I
COMPOSITION OF SOME COMMERCIAL FATTY ACIDS
PER CENT OF FATTY ACIDS PRESENT

	C ₁₄	C ₁₆	C ₁₈	C ₂₀	C ₂₂
A	5 to 8	28—33	23—28	18—23	15—19
B	5 to 8	25—30	20—25	20—23	18—25
C	—	22	78*	—	—
D	—	67	33*	—	—
E	—	60	40*	—	—

* These figures also include the oleic or unsaturated acids present. These range from 0.5% to 5%.

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PREPARATION OF GREASES

The lithium greases to be tested were prepared by slurring the lithium soap with the cold oil, raising the temperature of the mixture to 220°, 230° C. and holding this temperature while agitating until all the soap had dissolved and a clear solution had formed. The process usually required 15 to 30 minutes for a one-pound batch and was carried out in an open kettle with only slight darkening of the oil resulting. The greases were allowed to cool to room temperature with no special control of the rate since previous work had indicated that this variable was not critical for these oils. In all cases, 88% oil and 12% lithium soap was used, for this proportion gives a satisfactory grease and is well within the regular industrial formulation range.

PHYSICAL TESTS

After cooling and before any of the physical tests were run, the heavy cake of grease was broken up and run through a small hand operated grease gun fitted with a homogenizer nozzle. (Catalogue #7-042 Eimer & Amend N. Y. C.). This equipment provided a reproducible method of working the greases at an average rate of shear of 200 recip. seconds. After the

TABLE III
CHARACTERISTICS OF TEST OILS

Type	Viscosity	Flash Point	Pour Point	A.P.I. Gravity	V. I.
Paraffinic	343 sec/100° F.	445° F.	Zero	—	105
Naphthenic	305 sec/100° F.	385° F.	Zero	20	36

first pass through the worker, a penetration test was run using the standard ASTM penetrometer, this was designated as the initial penetration. This working process was then repeated with penetration determined after each pass until constant penetration, which usually required three to five passes, was reached. The increase or decrease in penetration between the initial and final determination was designated as Delta penetration. The Delta penetration values for paraffinic and naphthenic greases containing lithium soaps of 14 to 22 carbon atoms in length, are plotted in Figure I. The final or constant penetration values for this series are given in Figure II.

Bleeding tests were run on samples of each grease after constant penetration was reached using the method detailed in Army, Navy specification #ANG3 paragraph F-5c. Briefly, the test consists of measuring the percentage of oil which will separate from a weighed sample of grease placed in a small monel screen cone maintained at a constant temperature of 100° C. for 50 hours. Figure III summarizes the results of these tests.

Finally, dropping points were run using the standard ASTM equipment and procedure, and the results of these tests may be reviewed in Table IV.

ALTERATION OF PHYSICAL PROPERTIES

The results of the physical tests conducted provides conclusive evidence of the marked effect of the fatty acid chain length on the properties of the finished grease. In general, we observe that the Delta penetration is adversely affected and the final penetration of a grease greatly increased by acids above and below the molecular weight of stearic. It is interesting to note that the fatty acid giving optimum Delta and final penetration values, is dictated by the type of oil used. Thus,

a pure naphthenic stock requires stearic acid for best results, while a paraffinic oil produces the best grease when palmitic acid is used, so one must judiciously choose the fatty acid most suited to the particular oil or blend of oils employed if optimum results are to be realized.

Stability of a grease to bleeding, or separation of the oil phase is recognized as an important property and this is markedly affected by the fatty acid used in compounding. As demonstrated in Figure III, fatty acids with a molecular weight higher than stearic greatly reduce bleeding especially when a paraffinic oil is used, but as previously pointed out, these longer chain acids deleteriously influence Delta penetration and final penetration. We are then faced with opposing properties and are forced to accept a compromise.

Of the three factors studied, dropping point is least influenced by the fatty acid in the grease, but it is nevertheless obvious from the data in Table IV, that fatty acids of lower molecular weight than stearic, substantially increase the melting point. Again we face a compromise for while in some instances, higher dropping point is an advantage, the lower molecular weight acids, which give this property to a grease, not only accelerate bleeding, but cause poor work stability and decrease the final consistency of the lubricant.

UNSATURATED ACIDS

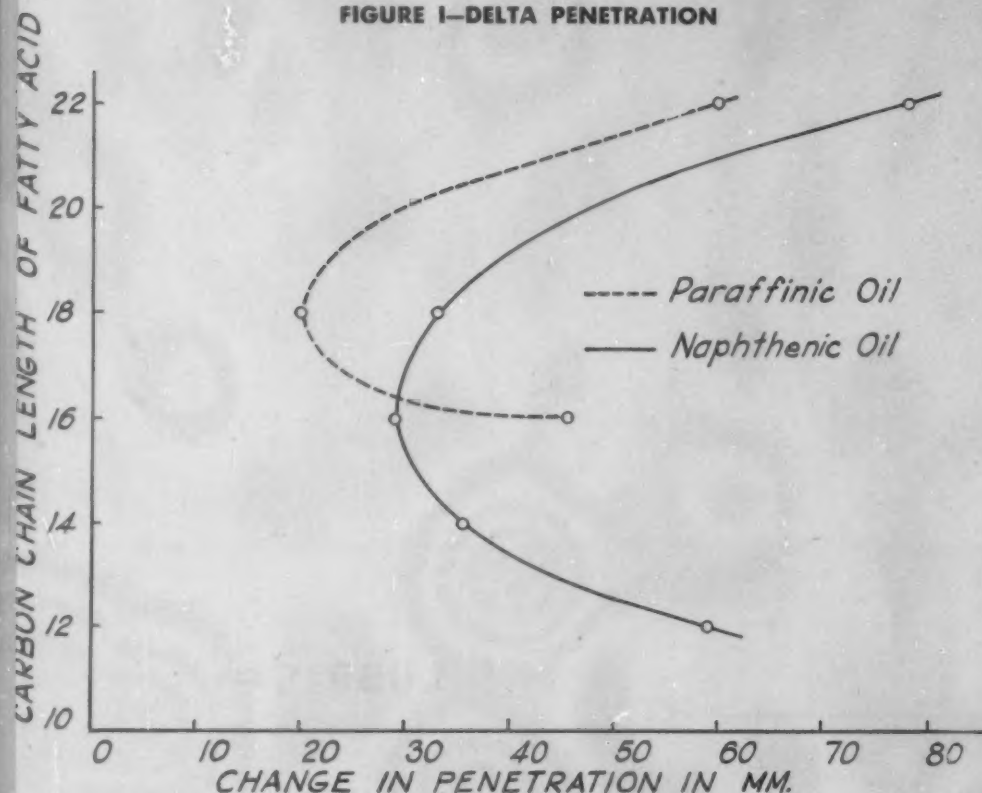
Heretofore, we have confined our discussion to the saturated fatty acids for these represent by far the bulk of the acids generally used in grease making. The unsaturated acids are generally not desirable, for, because of their instability, they tend to oxidize and polymerize readily, causing trouble in a grease. But, since most of the commercial fatty acids do contain small percentages of oleic, linoleic acid, etc., it is interesting to briefly note their

TABLE IV
A.S.T.M. DROPPING POINTS ON GREASES CONTAINING 12% LITHIUM SOAP
A.S.T.M. Dropping Points in °C for Lithium Greases from Various Fatty Acids

Fatty Acid Used	C ₈	C ₁₀	C ₁₂	C ₁₄	C ₁₆	C ₁₈	C ₂₀ C ₂₂	Ricinoleic Acid	Linoleic Acid	Oleic Acid
Naphthenic Oil	*	*	207°	202°	188°	187°	183°	153°	181°	182°
Paraffinic Oil	*	*	*	*	190°	188°	181°	153°	181°	181°

* The Lithium soaps of these fatty acids are not sufficiently soluble in the oils to form gels.

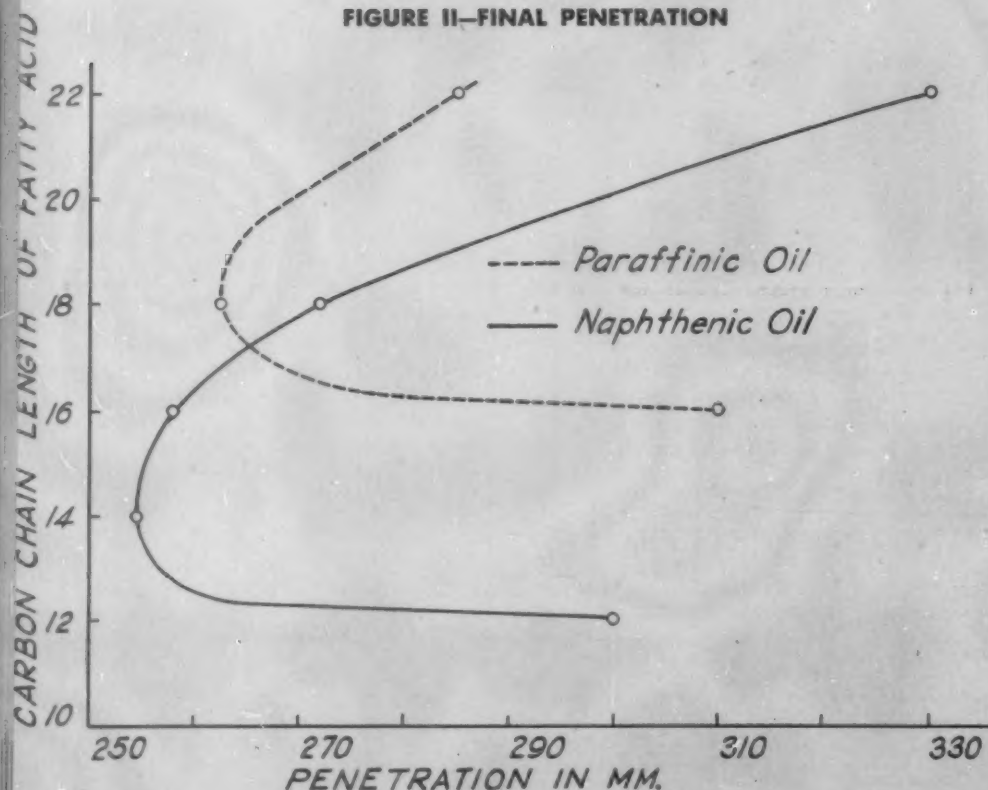
FIGURE I—DELTA PENETRATION



effect on a grease when they are present at a major portion of the fatty acid content. In Table IV, we see that in general, the unsaturated acids decrease the dropping point. It is generally conceded that they reduce the worked stability and seriously lower the consistency of a grease. They do however, decrease the bleeding and while this is an advantage, consideration of the over-all effect of unsaturated acids on the properties of a grease, influences one to admit that their absence is more desirable than their presence.

On the basis of this work, it appears conclusive that no one single fatty acid can be expected to produce a lithium grease of maximum value; rather, a judicious and carefully controlled blend of fatty acids is required to achieve optimum results. Considerable care in selecting the proper blend of fatty acids or their lithium salts should be exercised, dependent on the type or blend of oils from which the grease is to be compounded. Finally, it is evident that fatty acid blends or their lithium salts having minimum unsaturated acid content, should be chosen for production of stable, high quality greases.

FIGURE II—FINAL PENETRATION



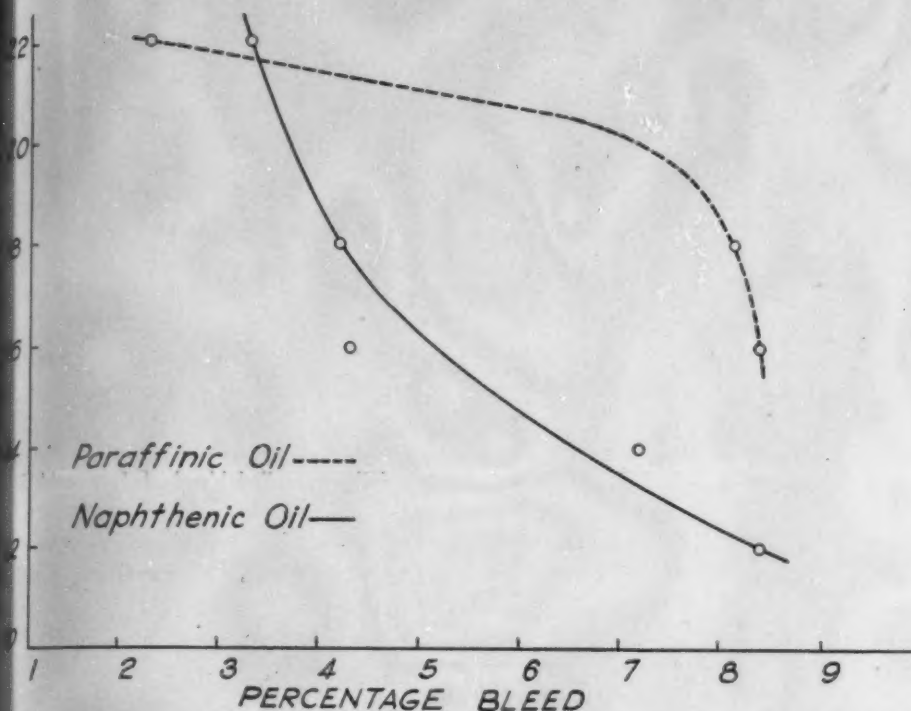
SUMMARY

It has been shown that the critical properties of a lithium grease are greatly affected by the carbon chain length of fatty acid used in its manufacture. Fatty acids with a molecular weight above and below that of a stearic were found to unfavorably influence penetration, bleeding and consistency stability. Naphthenic oils appear to produce better greases with lithium palmitate while paraffinic greases function better when made from lithium stearate. Unsaturated fatty acid lithium soaps did not produce satisfactory grease with either type of oil.

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2. American Society for Testing Materials—ASTM Designation D-566-40 T.
3. Earle, C. E.—U. S. Patents—2,274,674; 2,274,675; 2,274,676; 2,293,052; and Re-issue 22,299.

FIGURE III—ANG3a BLEED VALUES



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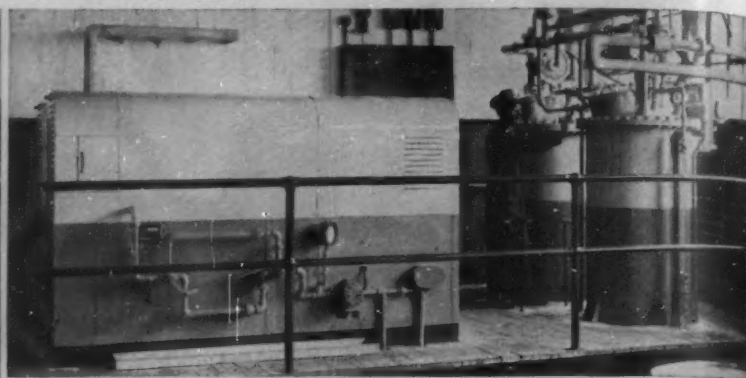
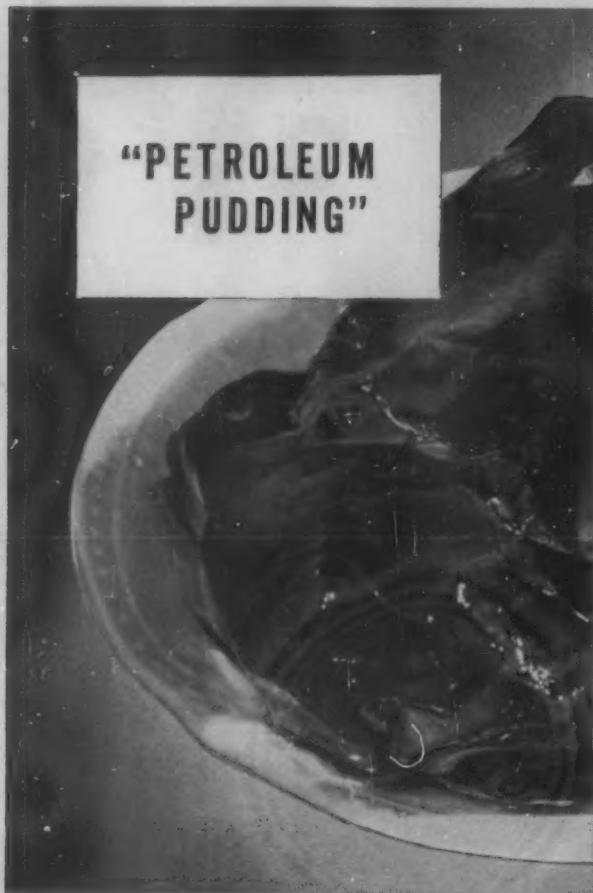
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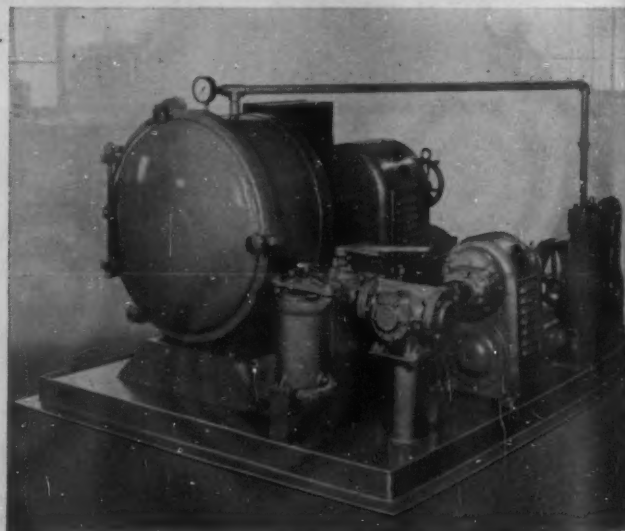
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